PROGRAM AND ABSTRACTS OF PAPERS

CITRUS RESEARCH CONFERENCE

December 8, 1964

Fruit and Vegetable Chemistry Laboratory
263 South Chester Avenue
Pasadena, California

Western Utilization Research and Development Division
Agriculture Research Service
UNITED STATES DEPARTMENT OF AGRICULTURE

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FOREWORD

This Citrus Research Conference is being held to bring to members of the citrus and allied industries in Southern California and Arizona the latest results of research on the chemistry, pharmacology, and technology of citrus fruits and their products carried on by the Utilization Research and Development Divisions of the Agricultural Research Service, U. S. Department of Agriculture. The following Divisions are participating in this year's conference.

Western Utilization Research and Development Division:
Western Regional Research Laboratory (Division headquarters), 800 Buchanan Street, Albany, Calif. 94710

Fruit and Vegetable Chemistry Laboratory, 263 South Chester Avenue, Pasadena, Calif.

Southern Utilization Research and Development Division:
U. S. Fruit and Vegetable Products Laboratory, 600 Avenue S, N. W., Winter Haven, Florida

U. S. Fruit and Vegetable Products Laboratory, 509 West Fourth Street, Weslaco, Texas

PROGRAM
CITRUS RESEARCH CONFERENCE
Tuesday, December 8, 1964

OXYGENATED TERPENES FROM ORANGES

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Western Utilization Research and Development Division
Western Regional Research Laboratory, Albany, California

Studies on stripping of volatiles from orange juice suggest that constituents of relatively high water solubility condense readily with the bulk of water in the first, water-cooled condensers. On the other hand, sesquiterpenoids which would be expected to be less water soluble pass on more readily to the brine-cooled condensers following in series. Thus a low degree of stripping should be adequate for relatively nonpolar constituents of moderate molecular weight range. The major esters of California Valencia orange juice were found to be ethyl 3-hydroxyhexanoate and ethyl acetate. The latter was inferred in the findings of Hall and Wilson (1925). Other compounds found in orange juice volatiles for the first time were: methyl 3-hydroxyhexanoate, ethyl sec-butyl ether, 2-methyl-3-buten-2-ol, 2-hexen-1-ol, n-butyraldehyde, 4-methyl-2-pentanone, and methyl ethyl ketone. Identification was by comparison of mass spectra with those of known compounds and matching gas liquid chromatography (GLC) retention times. Seven compounds previously tentatively identified by others only by GLC retention times were confirmed; namely, ethyl octanoate, octyl acetate, ethyl formate, 1,1-diethoxyethane, 1-propanol, citronellol and carvone. The C_5 to C_8 hydrocarbons may have come from solvents used in waxing fruits, biphenyl from fungistat treatment.

No mass spectra resembling the patterns for geraniol, nerol, citronellol, or isopulegol were observed at GLC retention time zones where these compounds should have appeared. These compounds were either absent or at such low concentrations that they were not detected. Oxygenated terpenes in a limonene distillate from orange oil were shown to include 1,8-cineol, linalool, isopulegol, neo-isopulegol, terpinene-4-ol and citral. This latter group of compounds was identified by GLC retention time, and infrared, nuclear magnetic resonance and mass spectra.

GRAPEFRUIT OIL SESQUITERPENES

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Investigation into the chemical composition of citrus peel oils has largely been motivated by a desire to understand the nature of flavor of these various oils. Although flavor is the cumulative sensation generated by the aggregate of chemical compounds present, precise understanding of flavor has lagged behind knowledge of the composition because of wide individual variability among naturally occurring organic compounds in flavor contribution. Grapefruit oil flavor has been particularly elusive to categorize in spite of the fact that for more than 30 years grapefruit oil has been known to consist of 90% limonene and numerous alcohols, aldehydes, and esters. Since the advent of gas chromatography, relatively complete characterization of the volatile chemical components, including those containing as many as 12 carbon atoms, still failed to resolve the ambiguity of grapefruit flavor, although better than 95% of the oil was accounted for. Among those components less volatile than the terpenes and oxygenated compounds then known to be present was a family of compounds containing 15 carbon atoms known as sesquiterpenes. These were investigated by gas chromatography.

The sesquiterpenes were concentrated by fractional distillation under vacuum at 70°-90° C./25mm. The remaining volatiles were rapidly distilled in a short path apparatus under high vacuum up to 180° C./mm Hg. The use of a molecular still for this operation is recommended. Hydrocarbons in the crude sesquiterpene distillate were separated by filtration in light petroleum solvent through a column of chromatographic alumina. Fractional distillation at 70°-90° C./5mm Hg afforded a sesquiterpene hydrocarbon fraction consisting of 8 principal components according to gas chromatography. This fraction, amounting to 1-2% of the oil, proved to be disappointing in its flavor contribution in that it was quite bland in odor and taste. However, because of its low volatility and solvent qualities, it may act as a neutral flavor fixative.

The oxygenated sesquiterpenes were collected by flushing the column of chromatographic alumina with ether and then fractionally distilling from 90°-150° C./1mm Hg. The largest fraction, b. p. 120°-140° C./1mm Hg, revealed a single major component when analyzed by gas chromatography. This component was the same one previously noticed by an industrial group and was subsequently

identified chemically and spectroscopically as nootkatone, a constituent of Alaskan Yellow cedar.

It had been noticed by the industrial group that there was a rough correlation between nootkatone abundance and grapefruit flavor intensity. Subsequent flavor study in consultation with a variety of persons experienced in citrus flavor evaluation has strengthened the initial tentative conclusion that nootkatone makes an important contribution to grapefruit flavor.

Once nootkatone was identified in the peel oil, it was also readily established as being present in peel-oil-free juice segments although in lower concentrations. An ether-petroleum extract of peel-oil-free grapefruit juice was prepared, chromatographed on alumina, distilled under high vacuum and collected from the gas chromatograph. An infrared spectrum identified the suspected peak as nootkatone. Similar analyses were performed on other commercial citrus oils, e.g., bergamot, lemon, lime, orange, and tangerine. Traces of nootkatone were found in each of these.

In sugar solution the taste of nootkatone is apparent at 40 p.p.m. The flavor is somewhat musty and astringent. Its abundance in grapefruit peel oil generally ranges from less than 0.1 to 1.5%.

DISTRIBUTION OF CAROTENOIDS IN CALIFORNIA MARSH SEEDLESS GRAPEFRUIT

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Mature grapefruit (Citrus paradisi Macfadyen, var. Marsh Seedless) has a distinctive yellow color which is due principally to carotenoid pigments located in the peel and pulp. Investigations of the carotenoids in immature and mature grapefruit have shown that both pigment mixtures are highly complex and strikingly different. During normal photosynthesis in the immature green fruit, the carotenoids are those required in the chloroplastin complex. The xanthophyll fraction becomes increasingly complex and the hydrocarbons undergo marked changes as the fruit ripens. Significant amounts of alpha- and beta-carotene were present in the peel of immature green fruit but diminished while the xanthophylls accumulated as the fruit ripened to a yellow color. No eta-carotene-like compound was detected in the pulp of immature green fruit. However, as the fruit matured to a yellow color, an eta-carotene-like component appeared and accumulated, becoming the major carotene component; and on further ripening or during prolonged storage, the eta-carotene-like compound diminished and zeta-carotene predominated.

RECENT DEVELOPMENT IN THE USE OF THE FOAM-MAT PROCESS FOR PRODUCTION OF CITRUS POWDERS

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Research concerning use of the foam-mat process for producing citrus powders has centered around four principal areas. The first of these is a study of the storage stability of orange and grapefruit powders made under different production conditions. This has included the study of different storage conditions as well. Secondly, research has centered around a study of certain characteristics of concentrates and the relative effect of these on drying behaviour and nature of the powder produced from them. A study of the factors affecting rate and ease of reconstitution has comprised the third area. Finally, a study of powders compared to the parent concentrates has been carried out on a more fundamental basis involving the use of chromatographic procedures.

In the determination of storage stability of orange and grapefruit powders, the use of antioxidants did not appear to enhance storage stability. To check the influence of oxygen more thoroughly, further investigations are presently underway using oxygen scavengers. In other storage studies, grapefruit powders have been stored up to 12 weeks without developing detectable flavor changes at 85°F., and have been stored for over 35 weeks at 70°F. without flavor changes. These flavor tests are based upon minimum discrimination of differences, and acceptability of the powders would go well beyond these storage times. Extensive storage tests have been started on both grapefruit and orange powders covering a wide range of temperatures, packaging materials, and packaging atmospheres.

Drying characteristics of concentrates were found very closely related to foam bubble size and structure. In studies of foaming agents and sizes of bubbles, several different type agents have been used successfully for the production of foams with good drying characteristics. A method of determining approximate average bubble size has been developed for use in evaluation of the suitability of foams for drying.

The powders produced from different type foaming agents have been compared for flavor, moisture content, drying characteristics, reconstitution, and general appearance. Myverol 18-00 was found to

be most beneficial for moisture release, probably due to formation of very fine bubble structure. From the standpoint of reconstitution ease and appearance, however, soya albumin (D-100) and Methocel-10 were found most effective.

Experimental drying runs have been carried out using several different additives to determine the effect on flavor and physical character of powders. These included the addition of orange "essence" to foam, before drying, and the use of orange concentrate to which peel oil had been added prior to canning. The addition of nootkatone to grapefruit concentrate before drying was also tried to determine the effect on flavor of the powder. Orange essense was found to be more effective toward improvement of flavor when added directly to the dried powder as a non-aqueous extract than when added to the foaming ingredients. The use of orange concentrate, to which peel oil had been added, was found to result in less efficient moisture removal during drying. This served to substantiate previous findings. The addition of nootkatone, isolated from grapefruit peel oil, to grapefruit concentrate prior to foaming, resulted in a considerable improvement in flavor of the grapefruit powder.

Studies on reconstitution have involved development of a densitizing treatment to increase bulk density of the powder. Temperature, release agents, and conditions for densitizing both orange and grapefruit powders have been developed, using a double-drum dryer adapted for the purpose.

Packaging studies using vacuum equilibration and carbon dioxide atmosphere have also been found to improve reconstitution and impart a better appearance to the reconstituted juice.

Analytical studies have been carried out on orange powders. The parent concentrate has been compared to orange powders stored at 0°F. possessing good flavor, and to powders which had been stored at 100°F. until strong "off-flavors" and "off-odors" developed. These studies have utilized gas, thin-layer and column chromatography techniques. They have included preliminary investigations of sugar and glycoside, acid, phenolic, and carotenoid fractions. There appears to be some relation between "off-odors" and carotenoid fractions. The acidic fraction has shown some indications of differences between the "off" powders and control powders. None of the fraction studies has indicated any differences between the parent concentrate and control powders.

In other recent developments, concentrates have been made from late season Valencia oranges. These experimental concentrates have been prepared with gross viscosities which varied over a wide range. They differ greatly in pulp content, serum viscosity, and pectin content as well. These concentrates are to be used for preparation of powders using the "crater" dryer.

The new "crater" dryer has been received, installed and is in operation. It is capable of producing good quality powders at a rate of approximately 25 pounds per hour. A few preliminary powders have been prepared but optimum conditions have not been fully developed yet.

AN INVESTIGATION INTO SEVERAL NEW CONSUMER PRODUCTS PREPARED FROM DESERT GRAPEFRUIT

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(Presentation by C. W. Kaufman)

Authorization for this project was not received until after this year's desert grapefruit harvest was completed. As a result, little more than planning and preparation has occurred in the last three months.

A program has been developed and some preliminary observations made on two types of juice product: (1) A "cloud" product with sugar, acid, bitterness, and color adjusted to most generally acceptable preference levels. (2) A "clear" product having the same flavor characteristics, but blended with other juices for color and flavor variations.

A second program has been outlined to explore ways of processing and pasteurizing a piece-formed grapefruit meat for use in salads, etc. The object of this program is to get an extended shelf life and lower preparation costs than products now on the market in dairy cases.

Industrial applications have been explored and a program mapped out for trying various grapefruit products in selected industrial uses.

POLYPHENOLICS OF GRAPEFRUIT

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Knowledge of the polyphenols of grapefruit is limited largely to those in the peel and peel oil fractions of the fruit. Very little is known about the edible portion of the fruit, the endocarp. Detailed knowledge of the endocarp should be helpful in improving existing grapefruit products and in developing new ones.

Chromatographic examination of the endocarp of desert grapefruit has now shown the presence of numerous polyphenolic constituents including coumarin derivatives, psoralens, hydroxycinnamic acid derivatives (probably esters and glycosides), and flavone and flavonone glycosides.

To facilitate the isolation and identification of the polyphenolic portion of these compounds extract from grapefruit endocarp was subjected to enzymic hydrolysis. The polyphenols released by this treatment were then separated by silicic-acid column chromatography, and identified from their melting points; infrared and ultraviolet absorption spectra; and paper chromatographic and electrophoretic comparison with authentic compounds. The principal polyphenols isolated were the flavanone, naringenin; the cinnamic acids, caffeic and ferulic acids; the coumarins, scopoletin; and the psoralen, bergaptol. Of these compounds bergaptol is the only one which occurs primarily as the free form in the endocarp before hydrolysis. Several other polyphenols were identified tentatively and work to confirm their identities is in progress. Work is also in progress on the isolation and identification of the esters and glycosides (or other derivatives) from which these polyphenols were released by enzymic hydrolysis.

REVIEW OF THE WESLACO LABORATORY'S RESEARCH ACTIVITIES ON CITRUS

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The historical development of citrus research at the Weslaco Laboratory is to be described briefly. The main efforts of the laboratory following its establishment in 1931 were directed toward aiding in the establishment of a fledgling citrus processing industry in the Lower Rio Grande Valley. As the number of processors in the Valley increased, the attention of the laboratory focused on a wide variety of citrus by-products and utilization of waste products. Following the development of citrus pulp dehydration, industrial utilization of citrus cannery waste ceased to be a problem.

Emphasis was switched to exploit the high degree of consumer acceptability of Texas Ruby Red grapefruit. The laboratory developed a method of pulp fortification by which a degree of redness characteristic of the natural fruit could be imparted to single strength juice. The pulp fortification process was further refined so that pulp from early season fruit which was high in color could be concentrated and preserved and then added to late season juice which was poor in color. The importance of the characteristic pigmentation of Ruby Red grapefruit is reflected in the quantitative and qualitative work which the laboratory has pursued on the carotenoids of processed juice. The carotenoid work has been expanded to make significant contributions to the understanding of the biosynthesis of carotenoids in citrus.

Although pulp fortification has helped to improve the color of Ruby Red juice, it has brought the problem of excess bitterness to the front. Weslaco has pursued two methods of debittering juice; one is enzymatic, the other involves physical adsorption of the bitter principles on selective resins. The laboratory has established a research contract with the Florida Citrus Experiment Station at Lake Alfred to investigate enzymatic debittering on an industrial scale. Some aspects of enzymatic debittering will be discussed.

The use of polyamide and polyvinyl pyrrolidone resins has shown promise in providing a rapid means of debittering juice on a laboratory scale. A public service patent application on the process has been submitted.

During the course of the work on pulp fortification and debittering, it became clear that the established chemical methods of determining the principle bitter component of grapefruit did not correlate well with subjective taste evaluations. Because of the need for a more reliable chemical test for several of the known flavonoid components of grapefruit, a research contract was negotiated with Dr. S. H. Wender at the University of Oklahoma. Some of the results of his group's research will be discussed. The Weslaco group is currently seeking to develop a better method of flavonoid analysis for industrial application.

The sequence of flavonoid production during the course of fruit development is being investigated with the aid of radioisotope studies. It is hoped that the cultural and climatic conditions under which a fruit has been grown may show a correlation with the flavonoid content and the bitterness of the processed juice. The utilization of cultural practices for improving product quality may ultimately be the most economical way of accomplishing this end.

Currently, the carotenoid investigations are continuing in cooperation with USDA Crops Research Division. One objective of this research is to determine the climatic conditions which initiate the decrease of lycopene and β-carotene in Ruby Red grapefruit.

RELATIONS BETWEEN THE TASTE AND STRUCTURE OF SOME PHENOLIC GLYCOSIDES

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Attempts to relate taste to chemical constitution are probably as old as chemistry itself. The subject is one of complexity, since the palate is extraordinarily sensitive to what seem to the chemist to be only minor variations in structure. Citrus fruits contain a number of flavanone glycosides in which the sugar component is either of the isomeric disaccharides, rutinose or neohesperidose. In earlier work it was shown that flavanones linked to rutinose (6-O-α-L-rhamnosyl-D-glucose) are tasteless, while those linked to neohesperidose (2-O-α-L-rhamnosyl-D-glucose) are bitter. The flavanone neohesperidosides often yield conversion products having an altered taste. For example, the chalcones and dihydrochalcones are usually sweet or bitter-sweet. Thus, naringin and neohesperidin yield intensely sweet dihydrochalcones, while poncirin yields a bitter-sweet dihydrochalcone. The dihydrochalcones made from the corresponding flavanone rutinosides are tasteless. The chemistry and structure-activity relations of this group of compounds will be reviewed.

A number of new dihydrochalcone neohesperidosides have been prepared. The synthesis and taste properties of these compounds will be discussed. An improved procedure for preparing the dihydrochalcones will be described and a brief summary given of their toxicology and metabolic fate in laboratory animals.

ISOLATION AND STRUCTURE OF SOME NEW CITRUS BITTER PRINCIPLES

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A new limonoid compound has been isolated from various citrus species. This material appears to be rather widely distributed in the genus Citrus and has been obtained on a preparative scale from seeds of:

C. aurantium Linn.	sour orange
C. sinensis (Linn.) Osbeck	navel orange seeds
C. grandis (Linn.) Osbeck	pummelo
C. paradisi	grapefruit
Poncirus trifoliata	
Ichang lemon (C. ichangensis X C. grandis)	

This new limonoid, $C_{26}H_{32}O_8$, m.p. 263-265°C, showed hydroxyl absorption in the IR and five C-methyl groups in the NMR. Thus, the new limonoid was more closely related to obacunone or nomilin than to limonin. Acetylation of the new material gave obacunone and nomilin which were separated by preparative thin layer chromatography. Since nomilin is known to eliminate acetic acid under acetylation conditions the new material appears to be deacetylnomilin. Circumstantial evidence indicates that deacetylnomilin is an intermediate in the biogenesis of limonin and that the sequence of steps in the latter stages of the biosynthesis of limonin is: deacetylnomilin → nomilin → obacunone → limonin. Examination of the literature suggests that deacetylnomilin might be identical with isolimonin, a limonoid isolated from various citrus species first reported by Koller and Czerny and later by Higby.

Workup of grapefruit seed meal extracts after removal of limonin by crystallization, by means of chromatography over alumina gave a new limonoid which proved to be identical with deoxylimonin. Deoxylimonin has previously been prepared synthetically from limonin. The orgin of deoxylimonin, which is non-bitter, probably arises from incomplete epoxidation during the biogenesis of limonin. Blocking this epoxidative enzyme system might offer a method for biological control of navel bitterness.

A third bitter principle has been isolated from ichang lemon. This polar material forms a mono- and diacetate and an anhydro-derivative. The NMR spectrum indicates it is closely related to limonin. A tentative structure is proposed for this material.

LIST OF PUBLICATIONS AND PATENTS*

Western Utilization Research and Development Division

Fruit and Vegetable Chemistry Laboratory
263 South Chester Avenue, Pasadena, California

CITRUS BITTER PRINCIPLES. I. A BIOGENETIC PROPOSAL FOR SEMIROUBACEOUS BITTER PRINCIPLES. Dreyer, David L., Experientia 20(6):297-298, 1964.

FLAVONOIDS OF CITRUS. VII. LIMOCITROL AND ISOLIMOCITROL. Gentili, Bruno and Horowitz, R. M., Tetrahedron 20:2313-2318, 1964.

NUCLEAR HYDROGEN-DEUTERIUM EXCHANGE IN RESORCINOLS AND RELATED COMPOUNDS IN WEAKLY ALKALINE SOLUTION. Hand, Elli and Horowitz, R. M., J. Amer. Chem. Soc. 86:2084-2085, 1964.

THE STRUCTURE OF 1, 3-NAPHTHALENEDIOL ANION. Hand, Elli S. and Horowitz, R. M., J. Org. Chem. 29:3088-3090, 1964.

RELATIONS BETWEEN THE TASTE AND STRUCTURE OF SOME PHENOLIC GLYCOSIDES. Horowitz, Robert M., Chap. 14 in "Biochemistry of Phenolic Compounds," ed. by J. B. Harborne, pp. 546-571, Academic Press, London and New York, June 1964.

STRUCTURE OF VITEXIN AND ISOVITEXIN. Horowitz, R. M. and Gentili, Bruno, Chem. & Indus. 12:498-499, March 21, 1964.

COMPOSITION OF CITRUS OILS. ISOLATION, IDENTIFICATION AND GAS CHROMATOGRAPHIC ESTIMATION OF SOME ESTERS AND ALCOHOLS OF LEMON OIL. Ikeda, R. M. and Spitler, E. M., J. Agr. & Food Chem. 12(2):114-117, 1964.

SESQUITERPENES. I. NOOTKATONE, A NEW GRAPEFRUIT FLAVOR CONSTITUENT. MacLeod, W. D., Jr. and Buigues,Nelida M., J. Food Sci. 29(5):565-568, 1964.

*Reprints are available at the addresses indicated; patents are available only by purchase at 25¢ a copy from the U. S. Patent Office, Washington, D. C.

Western Regional Research Laboratory
800 Buchanan Street, Albany, California

VOLATILES FROM ORANGES. I. HYDROCARBONS IDENTIFIED BY INFRARED NUCLEAR MAGNETIC RESONANCE AND MASS SPECTRA. Teranishi, Roy, Schultz, Thomas H., McFadden, W. H., Lundin, R. E., and Black, D. R., J. Food Sci. 28(5): 541-545, 1963.

NMR STUDY OF TRIMETHYLSILYL ETHERS OF FLAVONOID COMPOUNDS. Waiss, A. C., Jr., Lundin, R. E., and Stern, D. J., Tetrahedron Letters 10:513-518, March 1964.

RECENT DEVELOPMENTS IN COUMARIN CHEMISTRY. Stanley, William L., in "Aspects of Plant Phenolic Chemistry," Proc., 3rd Ann. Symp. Plant Phenolics Group of North America, Toronto, Sept. 6-7, 1963, pp. 79-103, 1964.

WHAT CAN BE DONE IN FLAVOR RESEARCH. Stanley, William L., Calif. Olive Assoc. Annual Technical Report 1963, pp. 67-76, 1964.

DEHYDROXYLATION OF CAFFEIC ACID BY RAT AND RABBIT CAECAL CONTENTS AND SHEEP RUMEN LIQUOR. Booth, A. N. and Williams, R. T. (St. Mary's Hospital Medical School, Paddington, London W2), Nature 198(4881):684-685, May 18, 1963.

DEHYDROXYLATION OF CATECHOL ACIDS BY INTESTINAL CONTENTS. Booth, A. N. and Williams, R. T. (St. Mary's Hospital Medical School, Paddington, London, W2) Proc., Biochem. Soc. 429th, June 27-28, 1963 bound with Biochem. J. 88(3):66P-67P, Sept. 1963.

SELECTIVE PARA- OR META-O-METHYLATION WITH CATECHOL O-METHYL TRANSFERASE FROM RAT LIVER. Masri, M. S., Robbins, D. J., Emerson, O. H., and DeEds, F. Nature 202 (4935):878-879, May 30, 1964.

METYLATION OF POLYHYDROXY-AROMATIC COMPOUNDS BY PAMPAS GRASS O-METHYLTRANSFERASE. Finkle, B. J. and Masri, M. S., Biochim. et Biophys. Acta 85(1):167-169, April 6, 1964.

PATENTS

(All patents are assigned to U. S. Government
as represented by the Secretary of Agriculture)

EXTRACTION OF FLAVORS. Stanley, W. L., Brekke, J. E., and Teranishi, Roy, U. S. Patent No. 3,113,031, Dec. 3, 1963.

Southern Utilization Research and Development Division

Fruit and Vegetable Products Laboratory
600 Avenue S, N. W., Winter Haven, Florida

FLOAT CHECK VALVE FOR USE WITH A WATER ASPIRATOR. Swift, Lyle James, Chemist-Analyst 52:23, 1963.

IDENTIFICATION AND DETERMINATION OF AN ADULTERANT IN CITRUS OILS. Hunter, G. L. K., J. Assoc. Off. Agr. Chemists 46(5):838-840, 1963.

CHANGES IN TOTAL CARBONYL CONTENT OF ORANGE JUICE AND CONCENTRATE DURING STORAGE. Senn, Vincent J., J. Food Sci. 28(5):531-536, 1963.

ISOLATION OF BETA-ELEMENE FROM ORANGE OIL. Hunter, G. L. K. and Parks, G. L., J. Food Sci. 29(1):25-26, 1964.

2, 4-p-MENTHADIENE. A NEW MONOTERPENE FROM VALENCIA ORANGE OIL. Hunter, G. L. K. and Brogden, W. B., Jr., J. Org. Chem. 29:498-502, 1964.

THE REARRANGEMENT OF LIMONENE AND CARVOMENTHENE EPOXIDES. Settine, R. L., Parks, G. L., and Hunter, G. L. K., J. Org. Chem. 29:616-618, 1964.

STRUCTURE OF YLANGENE. Hunter, G. L. K. and Brogden, W. B., Jr., J. Org. Chem. 29:982-983, 1964.

THE EFFECT OF BENZOIC, SORBIC, AND DEHYDROACETIC ACIDS ON THE GROWTH OF CITRUS PRODUCTS SPOILAGE ORGANISMS. Rushing, N. B. and Senn, Vincent J., Proc. Fla. State Hort. Soc. 76:271-276, 1963.

GENERAL APPLICATION OF NEAR-INFRARED MOISTURE ANALYSIS TO FRUIT AND VEGETABLE MATERIALS. Gold, Harvey J., Food Technol. 18(4):184-185, 1964.

A RAPID METHOD FOR ISOLATION AND IDENTIFICATION OF SESQUITERPENE HYDROCARBONS IN COLD-PRESSED GRAPEFRUIT OIL. Hunter, G. L. K. and Brogden, W. B., Jr., Analyt. Chem. 36:1122-3, 1964.

B-YLANGENE, A NEW SESQUITERPENE HYDROCARBON. Hunter, G. L. K. and Brogden, W. B., Jr., J. Org. Chem. 29:2100, 1964.

SHELF LIFE OF CHILLED ORANGE JUICE WITH HEAT TREATMENT AND PRESERVATIVES. Rushing, N. B. and Senn, Vincent,Jr., Food Technol. 18(8):112-114, 1964.

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P. O. Box 388, Weslaco, Texas

BIOSYNTHETIC RELATIONSHIPS BETWEEN CAROTENES. Purcell, A. E., Arch. Biochem. and Biophys. 105(3):606-611, 1964.

NOTES

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